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A Study on Ultrasonic Evaluation of Material Defects in Carbon/Carbon Composites

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It is desirable to perform nondestructive evaluation to assess material properties and part homogeneity because manufacturing of carbon/carbon (C/C) composites requires complicated and costly processes. In this work several ultrasonic techniques were applied to carbon/carbon composites for the evaluation of spatial variations in material properties that are attributable to the manufacturing process. In a large carbon/carbon composite manufactured by chemical vapor infiltration (CVI) method, the spatial variation of ultrasonic velocity was measured and found to be consistent with the densification behavior in CVI process in order to increase the density of C/C composites. Ultrasonic velocity and attenuation depend on a density variation of materials. Low frequency through-transmission scans based on both amplitude and time-of-flight of the ultrasonic pulse were used for mapping out the material property inhomogeneity. These results were compared with that obtained by dry-coupling ultrasonics. Pulse-echo C-scans was used to image near-surface material property anomalies such as the placement of spacers between disks during CVI. Also, optical micrograph had been examined on the surface of C/C composites using a destructive way.

Key Words: Carbon/Carbon Composites, Material Defects, Pitch Impregnation, Chemical Vapor Infiltration (CVI), Dry-Coupling Transducers, Ultrasonic Evaluation

1. Introduction

Composite materials (Hsu et al, 2002 ; Im, Sim and Yang, 2002) are attractive for a wide range of applications because of the advantage of very large strength-to-weight and stiffness-to-weight ratios. High performance engineering structures are being built with critical structural components made from composite materials. Especially, carbon/carbon (C/C) composites are one of the few materials that are suitable for structural applications at high temperature environments (e.g., above 2000℃) (Buckly, 1988) while maintaining strength and stiffness. Also, these composites possess a high resistance to thermal shock and are stronger at an elevated temperature. Thus, in the aerospace and vehicles industry, C/C composite materials are being used for aircraft brake disks, space shuttle rocket nozzles, exhaust cones, air

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inlet channels, connecting rod, engine piston and turbine rotor(Lewis, 1988). One such application is an aircraft brake disk. As compared to steel brakes, carbon/carbon brakes are lighter by about 40% and last twice as long as the steel brakes are in terms of the number of landings per overhaul (Awasthi and Wood, 1988). Aircraft brake manufacturers are therefore making brake disks and rotors out of carbon/carbon composites (Buckley, 1988; Fitzer, 1987). Aircraft brakes are critical components that serve multiple functions: they are the friction member, the heat sink elements, and the structural elements.

Compared with that for conventional composites such as Graphite/Epoxy, the manufacturing process for C/C composites is rather complex due to the multiple steps involved in the carbonization and densification cycles, which results in improved density and thermal conductivity. In one manufacturing approach, C/C composites are made by first producing pre-forms of carbon-fiber, which are then densified by a chemical vapor infiltration (CVI) process(Fitzer, 1987; Carbon/Carbon Composites, 1998). The CVI process is carried out on the C/C composites at high temperature (about 1000℃). The densification is repeatedly carried out until the matrix is so impervious to the vapor that no more carbon can be deposited on the fibers. At this point, the composites are quenched so that the matrix cracks due to thermal coefficient mismatch and thermal gradient methods. Also, under pressure in service, overcrusting and pore blockage may occur due to pressure gradient methods. The material is then heated again and densification is repeated. This process is repeated several times until a desired density is reached. Up to about three densification/carbonization may be required before the desired density is achieved. Porosity and cracks that often occur due to the mass loss and shrinkage of resin during the processing of carbonization have detrimental effects on the material properties of the C/C composites(Ko and Hone, 1992). To ensure product quality and structural integrity, nondestructive evaluation (NDE) methods (Stimson and Fisher, 1980; Nam and Seferis, 1992) are needed for inspecting carbon/carbon brake disks and rotors. Ultrasonic testing is capable of revealing material inhomogeneity and internal defects. More importantly, the velocity of ultrasonic waves is related to the elastic stiffness of the material in a direct relationship.

Ultrasonic C-scan imaging is an effective NDE technique used for material analysis and quality control in the industries. C-scan imaging in its most conventional implementation is used to map variations in ultrasonic echo peak amplitude that occur when scanning across a material part. Ultrasonic C-scan image provides quantitatively a two-dimensional view of a specimen in which differences in image contrast result from the objects interaction with an impinging ultrasonic wave (Hale, Hsu and Adams, 1996). Also, wave velocity is probably one of the most widely used quantities (Hsu and Hughes, 1992). When simply predicting the material property, it is very desirable to measure velocities on the sample surface. Such a method can be used as a nondestructive evaluation technique on structural components. In this work, several ultrasonic NDE techniques were applied in the evaluation of a developmental C/C composites. Through-transmission C-scans in an immersion setup based on both the amplitude and time-of-flight of the transmitted ultrasonic pulse were used for qualitative assessment of the material homogeneity in the plane of the disk. Ultrasonic velocity of longitudinal waves propagating in the thickness direction was measured at selected locations using elastomer-faced dry-coupling transducers. To correlate ultrasonic velocity with density variation and microstructures, a series of specimens were cut out along a radial direction for density measurement and microscopy. Ultrasonic pulse echo C-scans were used for detecting material anomalies near the surface of the disk. Finally, ultrasonic velocity and C-scan in the in-plane directions were measured to obtain the influence of fabric, chopped fiber and void for the severely cut-out pieces of C/C composites.

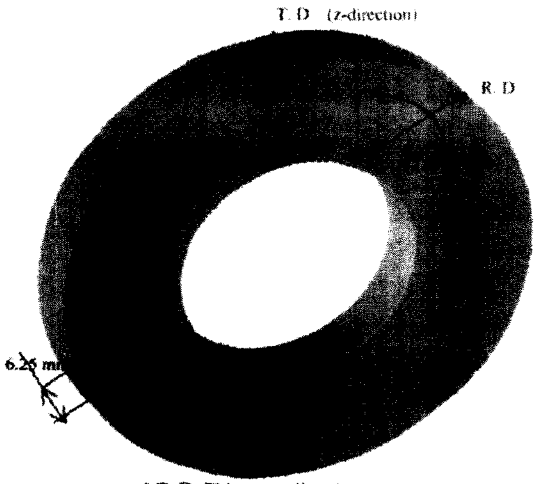
1.1 Curing process of C/C composites

The manufacturing method for carbon materials is similar to methods used for ceramic processes. Chopped fibers and Acelan fabric of solid particles (primary carbon part) are combined with coal tar pitch, which acts as precursor during the baking, i.e., carbonization treatment. The main disadvantages in the ceramic-like process method of carbon materials fabrication are the mass loss and shrinkage of precursor during a carbonization process. However, using the chopped fibers and coal tar pitch contribute to the reduction of the shrinkage to some degree. The first step in fabrication deals with the impregnation of carbon fabrics with the coal tar pitch. This is known as the as-cured step of the composites and then producing of molding packers. The composites are then subjected to carbonization, i.e., heat treatment at high temperatures. At this stage, the carbon of the composite increases, referred to as the carbonized composites. In this process, the pitch powder in the composites is pyrolyzed to form a carbonized matrix. The composite, at this stage of first carbonization, is highly porous and inferior in terms of its mechanical properties and density. Graphitization is performed at high temperatures (about 2000℃) to obtain the solid state transformation of metastable non-graphitic carbon into a graphite structure. The carbonization and graphitization steps in carbon-carbon composites are repeated two or three times. In order to improve the mechanical properties, it is then subjected to a densification process, in which the pores formed during pyrolysis are densified by using a chemical vapor deposition process.

2. Experimental Method

2.1 Sample configuration

In this test, two C/C composites (a C/C composite brake disk and C/C composite plate) were used. Test specimens were prepared with dimensions about 280×170×16 mm (outer dia.×inner dia.×thickness) and 290×220×12 mm (width×length×thickness) in a ring shape for the brake disks and C/C composite plate respectively. The C/C composite brake disk was manufactured from 22 doses of chopped fiber and 24 layers of fabrics impregnated with coal tar pitch. (Cabon/Carbon Composites, 1988) It was intended to be a symmetric laminate with respect to the mid-plane, but the actual brake is not symmetric. Three types of materials were used during fabrication. They are 8 harness satin weave fabric, the chopped fiber and the pitch powder. The pitch powder is used between the layers as a mechanical binder at elevated temperature. The inner fabric layers act as the load bearing part, while the outer chopped fiber layers are for the wearing (frictional) part. The most outer fabric layers are



* T. D: Thickness direction
*C. D :Circumferential direction
*R. D:Radial direction

Fig. 1 The C/C composite brake disk positions to be cut

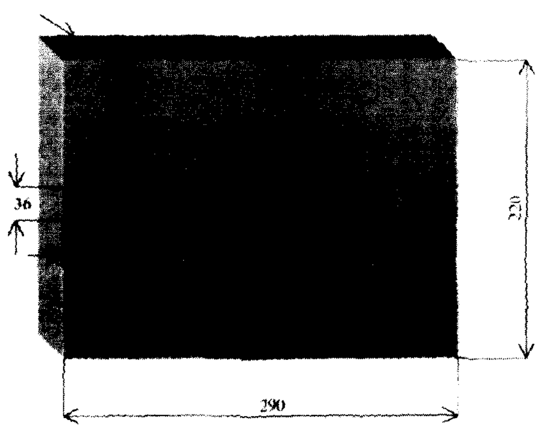


Fig. 2 The C/C composite plate positions to be cut

machined. The orientation of the chopped fibers can be assumed to be planar random. After finishing scanning the production C/C composites, we have cut the disk into eight pieces to compare destructive results with UT solutions with dimensions about 6.25×6.25×16 mm and about 36×36×12 mm respectively using a diamond saw as shown in Figs. 1 and 2.

2.2 NDE Techniques

The C/C composite disk used in this study (Carbon/Carbon Composites, 1998) was a ring-shaped disk 16 mm thick, 170 mm inner diameter, and 280 mm outer diameter and a C/C composite plate 290 mm width, 220 mm length and 12 mm thick was utilized. The composites consisted of a stack of cloth plies and chopped fiber layers and was densified by pitch impregnation and also by chemical vapor infiltration (CVI) at the end of fabrication process. All of the immersion scan and most of the dry-coupled velocity measurements were made with the disk intact. Afterwards, eight small blocks were cut out along a radius. The dimensions of each block were 6.25×6.25×16 mm as shown in Fig. 1. The densities of the blocks were determined from their individual weight and volume and the internal microstructures were examined using an optical microscope.

The through-transmission ultrasonic C-scans were conducted in an immersion tank using a SONIX scanning system shown in Fig. 3. A pair of 5 MHz, 6.35 mm diameter, unfocused transducers were aligned perpendicular to the disk and driven by a Panametrics 5052 pulser/receiver. Figure 4 shows schematic for experimental setup. The amplitude and time-of-flight of the (first arrival) transmitted pulse were used in generating the amplitude and time-of-flight images. The through-transmission measurement sampled the integrated material property in the thickness direction. In the transmission C-scan images, a larger amplitude is usually associated with better consolidated material with a lower attenuation, scattering, or absorption. The recorded time-of-flight in a transmission C-scan represents the sum of that in water and in the sample. Since the water path remains constant, a larger time-of-flight is associated with a lower velocity of sound in the sample. To probe the material properties near the surface, a low frequency transducer (5 MHz, 6.35 mm diameter, unfocused, respectively) was used in the pulse-echo mode. Amplitude C-scans were made using the front surface echo. Scan images were made using the overall peak-to-peak of the entire RF waveform after the mainbang. The image was then "equalized" for better contrast. Also, porosity in composites refers to the population of small voids in the material. These voids are the result of several times carbonization treatment and improper manufacturing process (e.g., inadequate CVI process) and have adverse effects on the stiffness and strength of the materials.

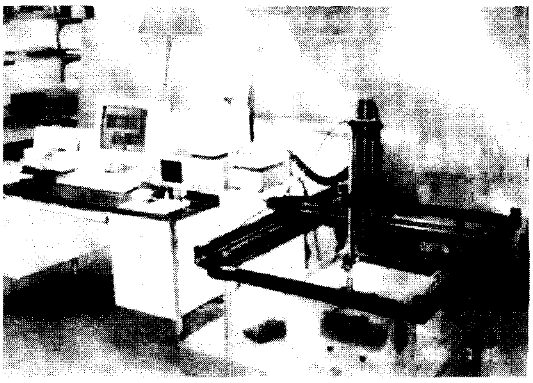


Fig. 3 SONIX scanning system for the ultrasonic measurement

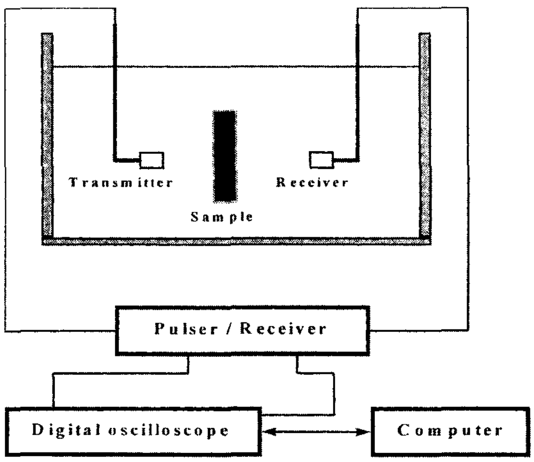


Fig. 4 Schematic for experimental setup (pulse-echo mode used in the study of C/C composites)

2.3 Velocity measurement

The ultrasonic velocity in the C/C composites was measured in the thickness direction, radial direction and circumferential direction specified at several points on the sample using dry-coupling ultrasonics (KD50-1, Ultran Lab.) as shown in Figs. 1 and 2. All measurements were made with a narrow bandwidth ultrasonic pulse transducers which have high power penetration and high sensitivity because C/C composites composing the chopped fiber, fabric and pitch makes the ultrasonic transmission difficult, and data were obtained in the time domain. In this work, contact and planar transducers of dry-coupling was used (see Fig. 5). To obtain a quantitative, accurate value of the ultrasonic velocity, a pulse-overlap method with dry-coupling transducers was used (Hsu, Liawand and Yu, 1994). These transducers contain an elastomer face layer and can be coupled by applying pressure. The measurement setup is shown in Fig. 5. To obtain the ultrasonic transit time through the sample, the difference in transmit time between two ultrasonic pulses was measured. The first pulse was transmitted through the reference piece but without the sample in plane and the record pulse was transmitted through the reference piece plus the sample. The thin rubber sheet was present in both cases to provide dry-coupling between the reference piece and the sample and to ensure that its own transit time was canceled out. These two pulses were stored in the memory of a LeCroy 9400 digital oscilloscope and their difference was obtained by shifting one pulse to overlap and match with the other pulse. The velocity was simply the sample thickness divided by the transit time through the sample. In this experiment, the transducers used were 1 MHz, 12.7 mm diameter (KD50-1, Ultran Lab.). The ability to store, shift, expand, invert and magnify the signals on the scope screen proved very convenient for performing the measurements manually. Figure 6 shows the typically overlapping pulses. The time-of-flight difference Δt was obtained by aligning the time cursor on each signal. The ultrasonic velocity is then given by $v=d/\Delta t$, where d is the sample thickness.

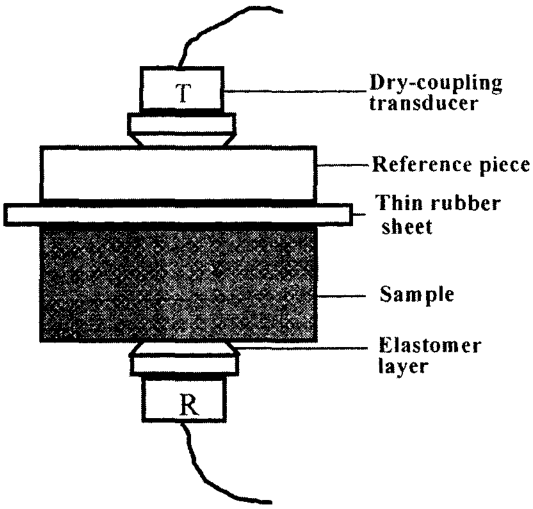


Fig. 5 Velocity measurement method using also a dry-coupling transducers. The transducers are 12.7 mm in diameter and have a center frequency of 1 MHz

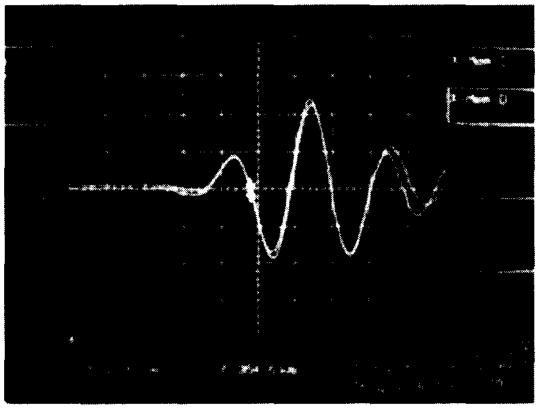


Fig. 6 The pulse-overlap method applied C/C composite

2.4 Density measurement

In order to verify the accuracy of the ultrasonic test procedure, we have cut the C/C composites into eight pieces with dimensions 6.25×6.25×16 mm to the radial direction of the C/C brake disk and with dimensions 36×36×12 mm on the greatest amplitude change region of all respectively and destructive analysis was conducted on the eight pieces cut from the C/C composites. Weight of each piece was measured using a Sartorius Analytic Balance (A200S, 1/10,000 g) and then density of each sample was obtained.

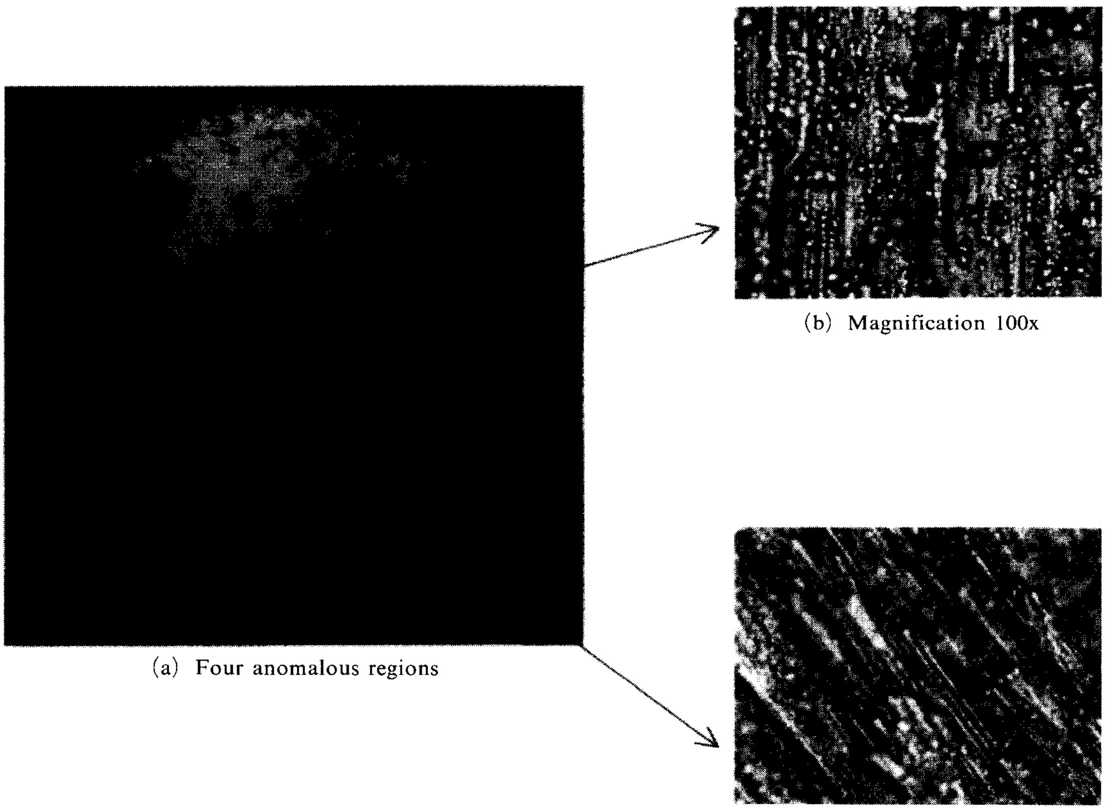
3. Results and Discussion

3.1 Anomaly detection

Through-transmission ultrasonic scans sampled the integrated material property over the entire thickness ; they were particularly sensitive to subtle anomalies near the surface. To detect minor material property variation near the surface, one could make use of the big amplitude variation of the front surface reflection (Hsu, Haghes and Patton, 1993). To examine the surface of the C/C brake disk, a 5 MHz, 6.35 mm diameter, unfocused immersion transducer was used to generate a C-scan image of the front surface echo amplitude. The resulting image, as shown in Fig. 7, revealed four circular rings equally spaced on the disk. Displayed are peak-peak amplitude of the front surface echo. These features were caused by the placement of "spacers" between adjacent C/C disks when a stack of disks was run through the final chemical vapor infiltration process. The spacers unavoidably impeded the infiltration and left a slightly less densified footprint underneath them. It was in fact confirmed that, for the disk in Fig. 7, the spacers used were solid of solid cylinders, which explained the circle-shaped features observed. Also Fig. 7(b) and (c) shows the surface of outside spacer and inside spacer location using optical micrograph method. Fig. 8 shows an image of C/C brake disk surface using thermal waves.

3.2 Through transmission

The amplitude and time-of-flight C-scan images of the through-transmission scan are shown in Figs. 9-10. Figure 9 shows that the transmitted signal was greater near rims of the disk diameter (id) and outer diameter (od) in some region. The time-of-flight image showed roughly that the



(a) Four anomalous regions

(b) Magnification 100x

(c) Magnification 100x

Fig. 7 Ultrasonic scan image shows four anomalous regions on the disk transit time was smaller near the id and od of the disk and larger between id and od. It should be pointed out that since the width of the disk (55 mm) was approximately 9 times that of the transducer diameter (6.35 mm), these amplitude and transit time variations in the radial direction were not just due to edge effects, but were attributable to material property nonuniformity The behavior of the ultrasonic transmission through the disk was consistent with changes in ultrasonic attenuation and velocity caused by density variation. Near the rims of the disk, the density was greater and the void content was lower; hence the attenuation was lower and the velocity was higher (Jeong and Hsu, 1995 and 1996). Between the id and od, the density was lower (higher void content); as a result, the attenuation was higher and the velocity was lower. There results were also consistent with the expectation of the pitch impregnation process. Because the pitch impregnation was most effective along paths between the plies, the regions near the id and od should be densified to a greater degree than the region between the id and od. Also, Fig. 10 shows an amplitude and B-scan images. At an A-A′ line there is an nonuniformity for an image. Amplitude of right area is higher; amplitude of left area is lower. These results were also consistent with time-of-flight of B-scan image. Therefore, it

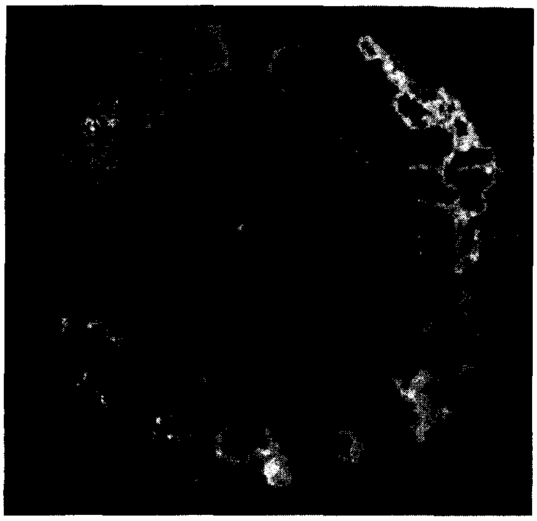


Fig. 8 Thermal waves image on the disk

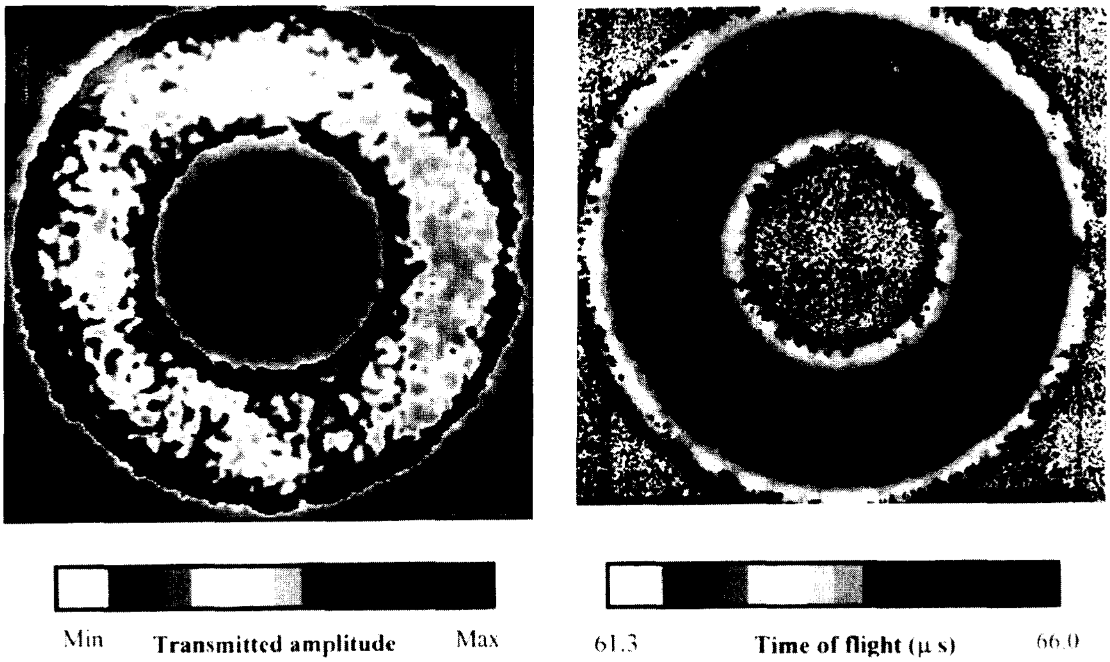


Fig. 9 Displayed are peak-to-peak amplitude and time-of-flight of through-transmission signal for the C/C brake disk. Transmitter and receiver are 5 MHz, 6.35 mm diameter and no focus transducer. Scan areas were 280×280 mm

is thought that these phenomena are attributed to an inadequate manufacturing process of C/C composites which involves repeated densification and pyrolysis (i.e., CVI process, etc.). Also, behavior of peak-peak amplitude variation is consistent with that of time-of-flight.

3.2 Ultrasonic velocity measurement

Using the method illustrated in Fig. 5, the longitudinal wave velocity in the thickness direction was measured at 18 locations shown in Fig. 11(a) and (b) respectively. Locations are shown with empty star (the same convention used in Fig. 12). The measured velocities varied between 1.78 mm/μs and 1.97 mm/μs. A plot of the velocity versus position, as shown in Fig. 12, revealed a highly regular pattern, the velocities at every point between the id and od were lower than those at the adjacent id and od. The measurements were repeated three times and the data are

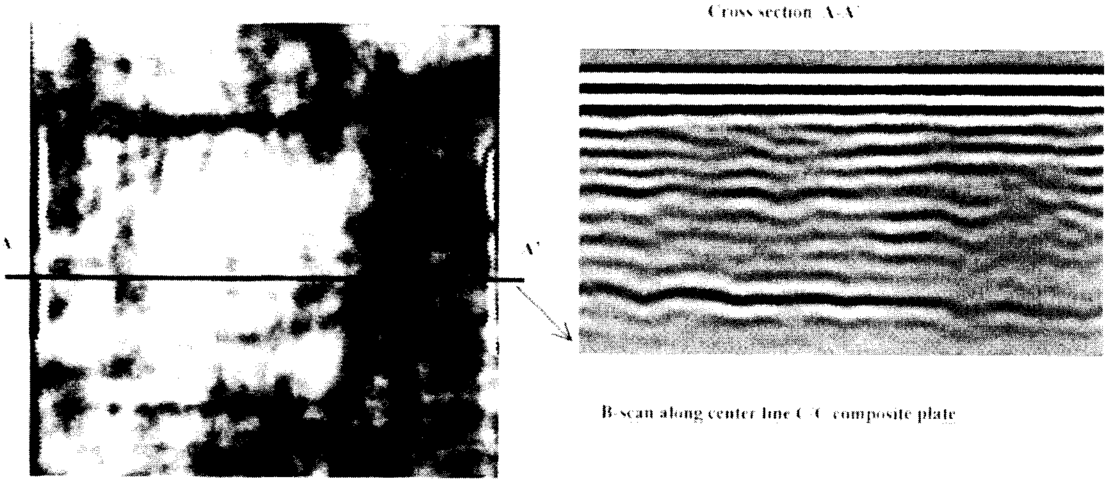
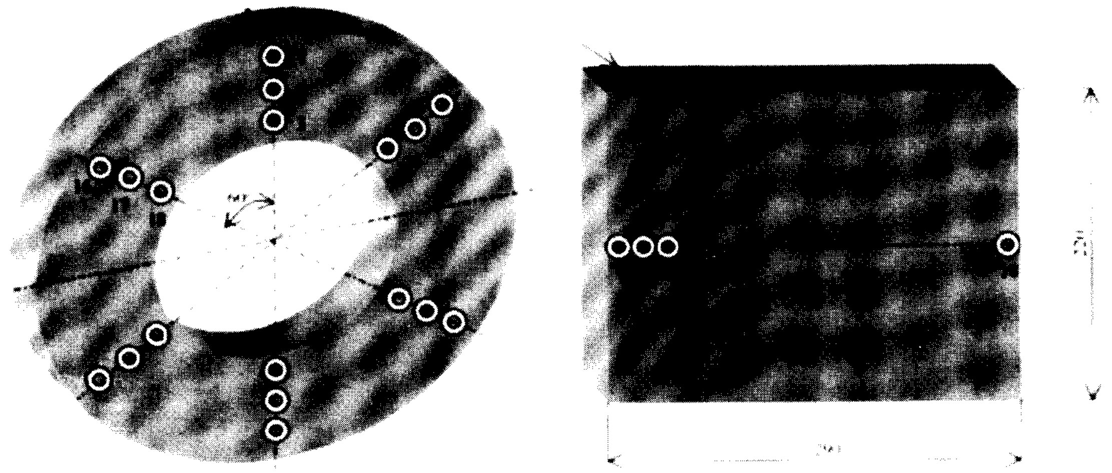


Fig. 10 Larger echo amplitude in C-scan image corresponds to shorter time of flight in B-scan image. Smaller echo amplitude in C-scan image corresponds to larger time of flight in B-scan image (Transducer: 1.5 MHz, 12.7 mm Dia. and no focus, Scan area is 304.8×279 mm)



◎ : Measurement positions on C/C Composite disk and plate

(a) C/C brake disk (b) C/C plate

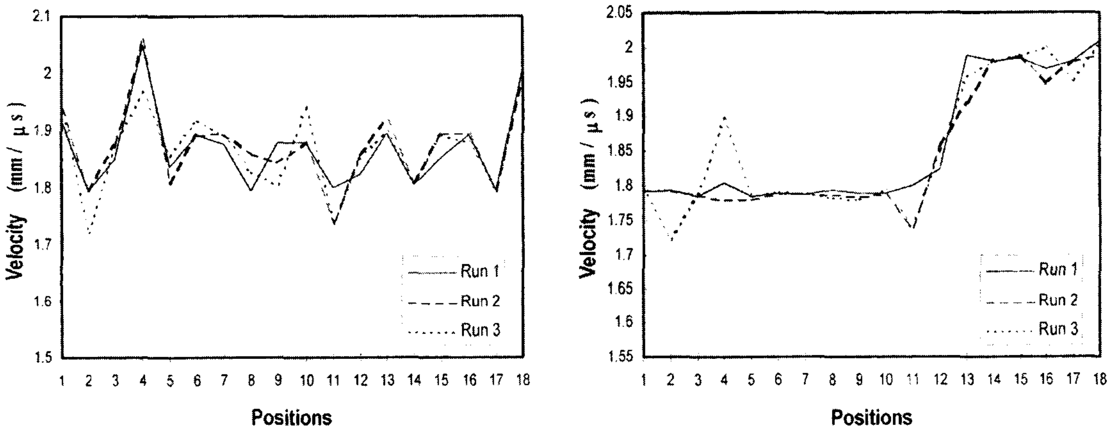
Fig. 11 Velocity measurement positions using dry-coupling transducer shown in Fig. 12 as Run 1, Run 2 and Run 3. The velocity data measured with dry-coupling transducers provided strong evidence of a consistent material property nonuniformity in the radial and transver direction in Fig. 12(a) and (b) respectively. Therefore, good results were obtained for both through-transmission and manually ultrasonic velocity measurements.

3.3 Destructive analysis

To verify that the above interpretation that the nonuniformities of the disk revealed by ultrasonic attenuation and velocity were caused by non-uniform impregnation and the resulting density variation, eight small sample were cut out along the radius direction. There samples, labeled No. 1 through No. 8 as shown in Fig. 1, were indivi-

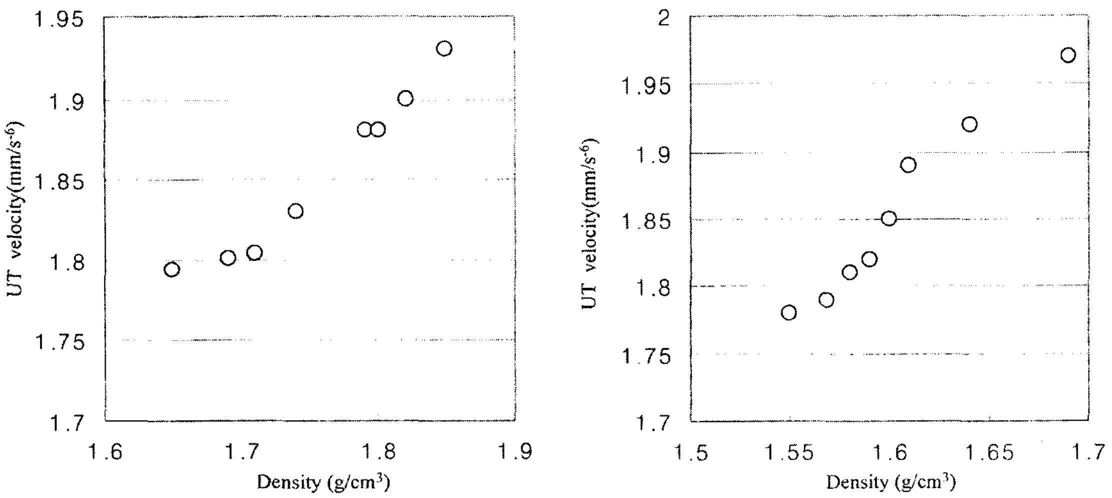
Table 1 Density variations for eight cut-out pieces

Sample No.	1	2	3	4	5	6	7	8	others
Density	1.85	1.80	1.82	1.65	1.69	1.71	1.74	1.79	C/C disk
(g/cm^3)	1.78	1.79	1.81	1.82	1.85	1.89	1.92	1.92	C/C plate



(a) C/C brake disk (b) C/C plate

Fig. 12 Relationship between measurement positions and velocity to the thickness directions



(a) Thickness direction of C/C composite disk (b) Thickness direction of C/C composite plate

Fig. 13 Relationship between density and velocity for the cut eight pieces dually weighed and their densities determined. The results are shown in Table 1. The density was clearly higher near the id and od, and lower in the middle region. The longitudinal wave velocity in the thickness direction was measured for each of the eight cut-out pieces and the results were correlated with the densities. Fig. 13(a) and (b) shows a clear correlation between ultrasonic velocity and density. For regions of a high degree of densification, the porosity content was low, the density was high, and the ultrasonic velocity was high. These regions had a greater stiffness. Conversely, regions of poorer densification had lower density, lower velocity and lower stiffness.

3.4 Cut-out pieces analysis

Figure 14 shows optical micrograph (Olympus, BH2) of one of eight cut-out pieces, which consisted of mainly chopped fibers and matrix at a higher half region of No. 1 cut-out piece which is

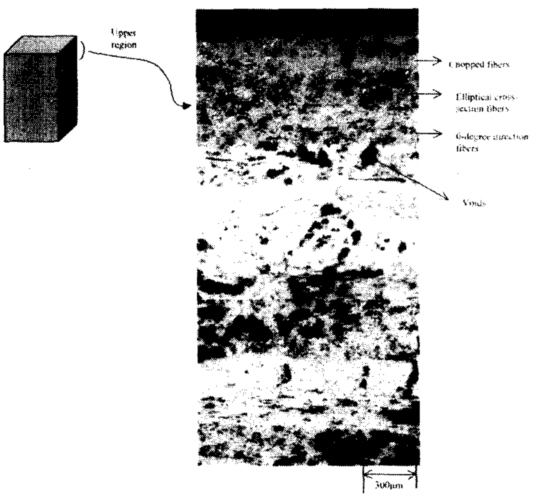


Fig. 14 Optical micrograph of cut-out eight pieces of C/C composite disk in upper region

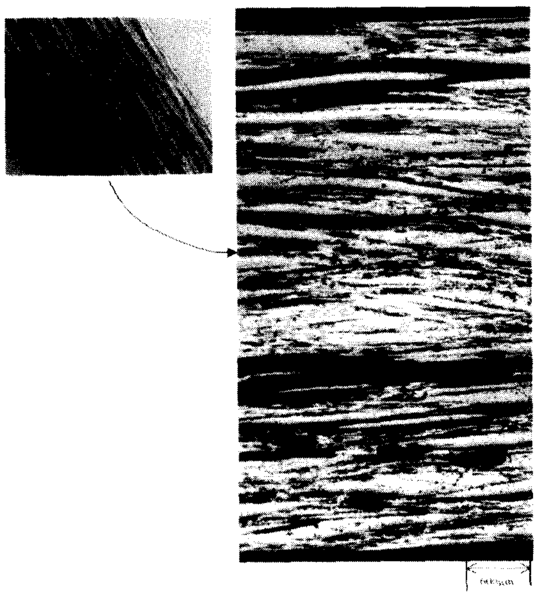
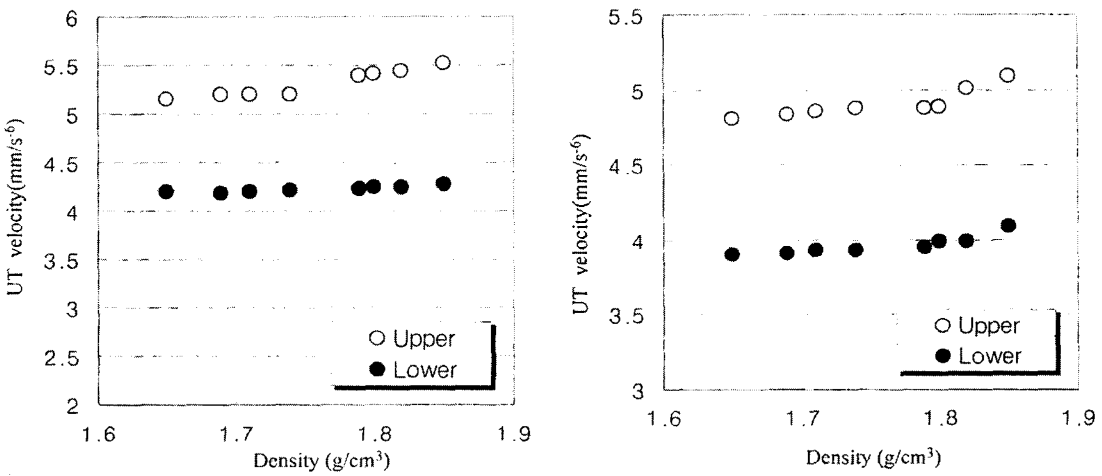


Fig. 15 Optical micrograph of C/C composite plate



(a) Radial direction of C/C composite disk (b) Corcumferential direction of C/C composite disk

Fig. 16 Relationship between density and velocity on the eight cut-out pieces to the radial and circumferential directions not symmetrically composed. Figure 15 shows optical micrograph at No. 1 cut-out piece with which is not symmetrically composed consisting mainly pores fibers and matrix. Figure 16 shows ultrasonic velocity in the radial and circumferential direction using the eight cut-out pieces. Higher velocity on the upper half region appears lower than lower half on both radial and circumferential direction images respectively. Velocity of radial and circumferential direction appears approximately three times as great as than that of thickness direction. Lower half had more chopped fiber and corresponded to lower velocity and lower attenuation. Upper half had more fabric and corresponded to higher velocity and higher attenuation. Therefore, the in-plane velocities in the radial and circumferential direction are essentially independent of density unlike the velocity in the thickness direction. However, the upper half and lower half had clearly different velocities due to their different contents of chopped fiber and fabric in C/C composites based on the optical micrograph methods.

4. Conclusions

To evaluate material properties and homogeneity of C/C composites, nondestructive evaluation methods are quite useful because of complicated and costly processes. The summaries in the present research are as follows:

(1) NDE pulse-echo C-scan method can provide the near-surface material property anomalies of placement spacers between disks during CVI. Also, relatively low frequency through-transmission scans were used for mapping out the material property variations.

(2) A dry-coupling ultrasonic technique is effective for predicting material properties as a comparison, and destructive analysis was conducted to verify the accuracy of the ultrasonic test procedure.

(3) It has been found that the ultrasonic velocity is different on both radial and circumferential directions for the cut eight pieces. The radial and circumferential direction velocity did not give good correlation compared to the thickness for the each piece density because chopped fibers are not uniform and stacking sequences are not symmetric.

(4) The most strong relationship was well observed between through-transmission C-scan images and dry-coupling ultrasonic results on the thickness direction in C/C composites disk and plate.

(5) A strong correlation was observed between through-transmission, dry-coupling ultrasonics and destructive results.

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